Oxygen contamination in liquid Argon: combined effects on ionization electron charge and scintillation light

WArP Collaboration

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Abstract

A dedicated test of the effects of Oxygen contamination in liquid Argon has been performed at the INFN-Gran Sasso Laboratory (LNGS, Italy) within the WArP R&D program. Two detectors have been used: the WArP 2.3 lt prototype and a small (0.7 lt) dedicated detector, coupled with a system for the injection of controlled amounts of gaseous Oxygen.

Purpose of the test with the 0.7 lt detector is to detect the reduction of the long-lived component lifetime of the Argon scintillation light emission at increasing O_2 concentration. Data from the WArP prototype are used for determining the behavior of both the ionization electron lifetime and the scintillation long-lived component lifetime during the O_2-purification process activated in closed loop during the acquisition run. The electron lifetime measurements allow to infer the O_2 content of the Argon and correlate it with the long-lived scintillation lifetime data.

The effect of Oxygen contamination on the scintillation light has been thus

measured over a wide range of O_2 concentration, spanning from $\sim 10^{-3}$ ppm up to ~ 10 ppm. The rate constant of the light quenching process induced by Oxygen in LAr has been found to be $k'(O_2)=0.54\pm 0.03$ $\mu s^{-1}ppm^{-1}$.

Contents

1   Introduction

Ionization in liquid Argon (LAr) is accompanied by scintillation light emission. Charged particles interacting in LAr create free electrons (e^-) and excited Ar molecular states (Ar_2^*) which produce scintillation radiation through de–excitation processes [1]. Therefore, free electron separation and light emission are the two features that characterize the use of LAr as active medium [2]. The two-phase (liquid/gas) technology developed by the WArP Collaboration

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is based on the simultaneous collection of both signals [3].

The two processes are complementary and their relative weight depends on the strength of the electric field (EF) applied to the active LAr volume [2]. The free electron yield (from ionization) rises with the field value while the photon yield (from scintillation) decreases. For a minimum ionizing particle (*mip*) at low fields (e.g. 0.5 kV/cm) the free electron yield is $Y_{ion} = \sim 2.9 \times 10^4$ e^-/MeV [4]. At the same EF the photon yield is also high ($Y_{ph} =\sim 2.4 \times 10^4$ γ/MeV) [5], for more densely ionizing particles is even higher. The light emission is characterized by a narrow–band intrinsic spectrum (around 127 nm) in the deep ultra-violet region (Vacuum-UV, VUV) [6],[7] and by a two-component exponential decay (short and long), characterized by τ_S in the 2 ns to 6 ns lifetime range and τ_T in the 1.1 μs to 1.6 μs range respectively [6],[8].

Tiny fractions of impurities (O_2, N_2, H_2O and $CO+CO_2$) diluted at ≤ 1 ppm level in LAr are usually reported in commercially available Argon (best grade), due to the industrial process of air separation. These impurities may impair the detector performance by significantly reducing the amount of both charge and light available from ionization events in LAr. This is due to various reactions involving different types of impurities, the most harmful of which being Oxygen. Since the pioneering R&D work by the Icarus Collaboration [9], Argon purification systems (Oxygen reactants and molecular sieves) are known to be very effective in reducing the O_2 contamination and also H_2O and $CO+CO_2$ to a negligible level (down to ≤ 0.1 ppb).

Detailed studies of the effects of Oxygen contamination on ionization electrons have been performed in the past. On the other hand, the effects on LAr scintillation light have been less investigated[1] . Purpose of the present work is to elucidate this last aspect, eventually by combining with available data on free electron trapping by O_2.

2 Residual Oxygen contamination in liquid Argon

Two are the effects attributed to O_2 contamination in LAr: (1) the ionization electron attachment and (2) the scintillation light quenching. Characteristics of these processes are briefly summarized here below.

2.1 Ionization electron attachment

The main limitation for a full collection of the free electron charge in LAr is due to the residual concentration of electro-negative molecules. In fact, electron attachment processes to electro-negative impurities like O_2 are active:

$$e^- \ + \ O_2 \ \rightarrow O_2^- \tag{1}$$

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[1] A study of the effects of air traces (N_2+O_2) in Argon gas can be found in Ref.[10].

Under the assumption that the free electron concentration $[e^-]$ produced by ionizing events is smaller than the impurity concentration [2] $[O_2]$, the free electron concentration decreases in time as:

$$\frac{d\,[e^-]}{dt} \;=\; -\,k_e\,[O_2]\,[e^-] \quad\Rightarrow\quad [e^-](t) \;=\; [e^-](0)\,e^{-t/\tau_e} \tag{2}$$

where the *electron lifetime* τ_e is defined as:

$$\frac{1}{\tau_e} \;=\; k_e\,[O_2] \tag{3}$$

The value of the *rate constant* k_e depends upon the drift field applied to the active LAr volume. The reference value [3] at EF=1 kV/cm is k_e= 5.5×10^{10} lt moles^{-1} s^{-1} [11] (equivalent to 1.9 ppm^{-1} μs^{-1}).

2.2   Scintillation light quenching

Residual O_2 contamination leads also to a substantial reduction of the scintillation light intensity. This may be attributed to a quenching process in two-body collision of O_2 impurities with Ar_2^* excimer states:

$$Ar_2^* \;+\; O_2 \;\rightarrow\; 2Ar \;+\; O_2 \tag{4}$$
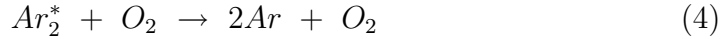
This non-radiative collisional reaction is in competition with the de-excitation process leading to VUV light emission. As a result, a sensitive quenching of the scintillation light yield is expected, mainly in the slow-component amplitude. In fact, the quenching process leads to a decrease of the excimer concentration $[Ar_2^*]$, while the contaminant concentration $[O_2]$ stays constant in time. To a first approximation, also for this case a first order rate law can be assumed, characterized by the rate constant k':

$$\frac{d\,[Ar_2^*]}{dt} \;=\; -\,k'\,[O_2]\,[Ar_2^*] \quad\Rightarrow\quad [Ar_2^*](t) \;=\; [Ar_2^*](0)\,e^{-t\,k'\,[O_2]} \tag{5}$$

The slow component lifetime of the scintillation light emission results to be effectively decreased, depending on the O_2 concentration:

$$\frac{1}{\tau_T'} \;=\; \frac{1}{\tau_T} \;+\; k'\,[O_2] \tag{6}$$

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[2] The concentration units in use here are ppm, *parts per million (atomic,* often indicated as ppma) and ppb, *part per billion.* These indicate the ratio between the number of interesting elements (in our case O_2 molecules) to ordinary elements (Ar atoms).

[3] Other measurements of k_e are reported in literature [12] or under the value of correlated variables [13],[14]: these agree within $\sim \pm25\%$. At null field a value of 1×10^{11} lt moles^{-1} s^{-1} is also reported in [11], corresponding to an electron lifetime of $\sim$ 3 ms at 0.1 ppb of Oxygen concentration.

To our knowledge, no data are reported in literature regarding the actual value of the Oxygen rate constant k' and its measurement is one of the goals of the present study.

3 WArP 2.3 lt prototype test

The WArP prototype is operated underground at the Gran Sasso INFN Laboratory (LNGS), in the tunnel connecting HallA to HallB. Data reported here refer to the Aug. '07 run. The detector is a two-phase drift chamber, with a lower liquid Argon volume and an upper region with Argon in the gaseous phase, both viewed by the same set of photo-multipliers (PMTs) [15]. Free electrons generated by ionization in the liquid are drifted by means of an electric field to the liquid-gas interface, where they are extracted through the boundary and detected by the proportional scintillation light generated by the electrons accelerated in a high electric field.

The drift volume, 7.5 cm long, is delimited by a 20 cm diameter stainless steel cathode and by a system of field-shaping electrodes that generate very uniform electric drift fields in the 1.87 lt sensitive volume ($\simeq$ 2.3 lt total volume). A grid (g1) placed just below the liquid level closes the uniform drift field region, while two additional grids (g2 and g3 from bottom to top) are placed in the gas phase. In normal data taking conditions, the chamber is operated with a drift field of EF = 1 kV/cm.

Four 12-stage 3" photo-multipliers (PMTs), manufactured in order to operate at LAr temperature and placed at about 4 cm above the last grid, detect both the primary scintillation in the liquid (the S1 signal) and the proportional scintillation light in gas phase (S2 signal). Sensitivity to VUV photons emitted by the scintillating Argon is achieved by coating the photo-multiplier window with an appropriate compound, i.e. Tetra-PhenylButadiene (TPB), which acts as a fluorescent wavelength shifter of the VUV scintillation light to the photo-multiplier sensitive spectrum. In order to improve the light collection efficiency from the drift volume, a high performance diffusive reflector layer with TPB deposit surrounds the inner drift volume and the gas volume between the top grid and the PMTs.

The system is contained in a stainless steel, vacuum-tight cylindrical vessel. The whole container is cooled down to about 86.5 K by an external liquid Argon bath. This set-up ensures in the inner container a constant absolute pressure few mbar above the external atmospheric pressure (P$\simeq$ 900 mbar).

Gaseous Argon (GAr) for filling is the best grade 6.0 (99.99990%) commercial Argon, with impurity concentration below 1 ppm (of which $\leq$ 0.2 ppm of O_2 and $\leq$ 0.5 ppm of Nitrogen, N_2, according to specifications). At filling, GAr is flushed through a standard Oxysorb/Hydrosorb cartridge for initial removal of O_2 and H_2O. An Argon recirculation system is implemented working in closed loop and providing a further continuous re-purification of the Argon contained in the chamber.

The anode signal from of each PMT is integrated (shaping time 120 μs) and

sent to a 10 bit flash ADC with 100 MHz sampling frequency. At each trigger the memory buffer is then recorded to disk.

3.1 Electron lifetime measurement

The amount of free e-charge released by ionization events in the liquid phase can be determined by the pulse-height of the secondary signal (S2). This is proportional to the number of free electrons extracted in the gas phase. According to the reaction of Eq.2, electron attachment to electro-negative O_2 (or O_2-equivalent) molecules diluted in LAr may occur during the drift up to the liquid-to-gas separation surface. Therefore, the amount of charge extracted results to be exponentially lessened at increasing drift distance of the ionization event from the surface.

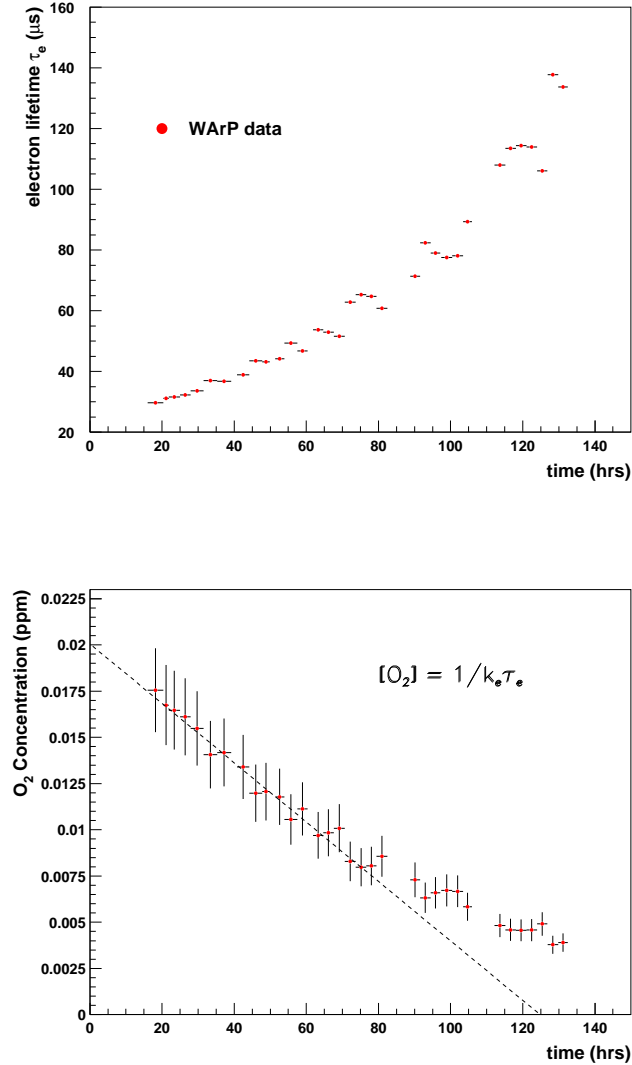For each recorded ionization event the drift time can be determined as the

Fig. 1. *Time evolution of τ_e [Top] and O_2 concentration [Bottom] in the WArP 2.3 lt chamber during Argon purification process.*

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time interval between S2 and S1 signals. The distribution of the S2/S1 pulse-height ratio vs drift time for the events [4] recorded in a defined time slot shows an exponential decay trend, with characteristic time constant (the *electron lifetime* τ_e) to be determined by fitting. The corresponding O_2 concentration can be inferred from Eq.3, through the k_e value at the drift field of operation (assumed as known [11], 1.9 ppm^{-1} μs^{-1}).

Measurements performed in successive time slots (of about 3 hrs) after filling, during Argon recirculation through the purification system, indicate that the electron lifetime progressively increases (i.e. the Oxygen concentration reduces) up to the limit of sensitivity of the method employed (≥ 200 μs). The results are shown in Fig.1, [Top] the measured τ_e and [Bottom] the corresponding [O_2] behavior as a function of run time after filling. The decrease of the O_2 concentration initially follows a linear behavior in time; this allows to extrapolate the initial O_2 contamination of the Argon in the chamber. This is found to be around 20 ppb. It comes from the reduction of the original O_2 content in the 6.0 grade GAr (≤ 0.2 ppm), due to the purification procedure at filling time, partially balanced by contributions from unavoidable material outgassing inside the detector and from the residual leak rate of the filling system.

3.2   Long-lived scintillation lifetime measurement at low O_2 concentration

The PMT signal with the WArP 2.3 lt detector is integrated and digitized with 10 ns sampling time. Pulse shape analysis of the recorded primary signals (S1) allows the reconstruction of the main features of the scintillation light emission following ionization events in LAr. In particular, taking into account the shaping time of the charge amplifier in use, the long-lived component decay time (τ_T) can be determined by a fitting procedure of the signal shape. The fit is applied to the waveform obtained as the average of the signals recorded in each time slot defined in Sec.3.1. In Fig.5 [Left] an averaged waveform (from data in a given time slot) is shown with the fitting function superimposed.

The [O_2] mean level of the corresponding time slot has been inferred from the electron lifetime analysis (Sec.3.1). The correlation of the measured long-lived decay time (τ_T) with the actual Oxygen concentration is reported in Fig.2 for the full set of data.

   The correlation is rather weak and the almost constant τ_T behavior in the range below $\simeq 20$ ppb indicates that at this (low) contamination level scintillation emission is unaffected (or only marginally affected) by the quenching process of Eq.4. Dedicated measurements at higher O_2 concentrations are needed to find appreciable effects (see Sec.4.1).

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[4] These are background events mainly from beta emission of ^{39}Ar in natural Argon.
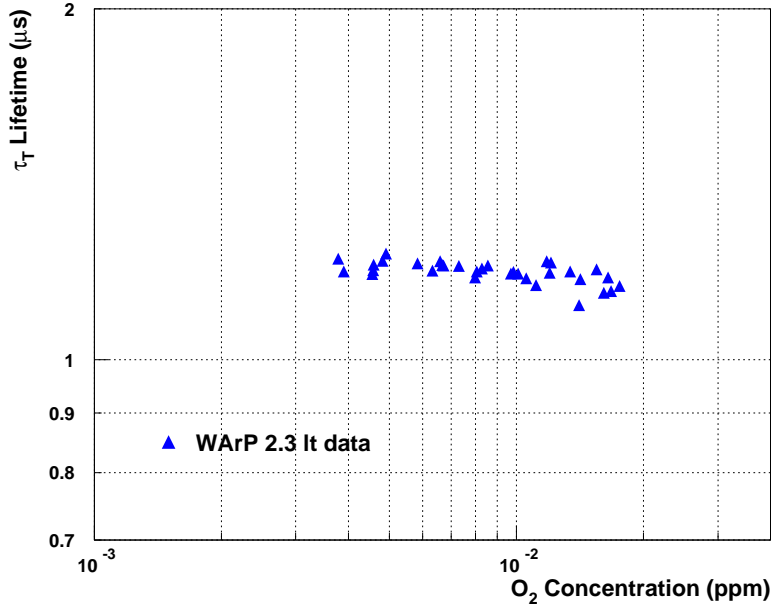
Fig. 2. *Long-lived scintillation lifetime from the fit of the average waveforms (WArP 2.3 lt data) vs Oxygen concentration (inferred from the electron lifetime analysis).*

4 Oxygen contamination test with a 0.7 lt detector

A detector dedicated to the study of Oxygen contamination effects on LAr scintillation light has been assembled and operated at the LNGS external facility ("Hall di Montaggio", Jan.-Apr. '07). It consists of a cylindrical LAr cell in PTFE (h=12 cm, $\varnothing$=8.5 cm internal dimensions, wall thickness 0.5 cm) containing about 0.7 lt of LAr (about 1 kg of active mass), viewed by a single 2" PMT mounted on the open top end of the cell, of the same type of those in use with the WArP prototype (including the wave-length shifting coating of TPB). A reflector layer also covered with TPB surrounds the internal walls (side and bottom) of the PTFE cell.

The detector is housed in a stainless steel cylindrical chamber, closed at both ends by vacuum tight flanges. The internal volume of the chamber is about 6.5 lt and it contains, after filling, a total amount of 3.0 lt of LAr (including the LAr cell active volume at its bottom). The chamber is immersed in a LAr bath of a stainless steel open dewar, to liquify and keep at stable temperature the LAr internal volume (T$\simeq$ 86.7 K, P$\simeq$ 910 mbar).

A transfer line for the Ar filling and for the injection of controlled amounts of O_2 (from a minimum of about 50 ppb per injection) has been connected to the LAr chamber. Gaseous Argon (GAr) for filling is the best grade 6.0 commercial Argon, as for the WArP 2.3 lt run. The GAr is flushed through a Hydrosorb/Oxysorb cartridge for "partial" removal of O_2 and H_2O positioned

along the GAr filling line. No additional recirculation/O_2-repurification system is implemented with this detector.

The PMT anode signal is directly read–out by a fast *Waveform Recorder* with sampling time of 1 ns over a full record length of 10 μs.

After the first run at 0 ppm (i.e. no additional O_2 injection to the initial purified 6.0 Argon), an experimental test was performed by adding progressively controlled amounts of O_2 (total content 60 ppb, 300 ppb, 600 ppb, 2 ppm, 5 ppm, 10 ppm), waiting for few hours and exposing the LAr cell to γ-sources after each contamination.

More information about the detector and details of the data treatment can be found in [17] where results from another test of LAr contamination with Nitrogen are reported.

4.1 Long-lived scintillation lifetime measurement at high O_2 concentration

Waveform recording of the direct PMT signal (no charge amplifier stage) allows for a detailed study of the LAr scintillation light emission, in particular of the individual exponential components (relative amplitude and decay time). To this purpose the average waveforms, from each run taken in presence of incremental O_2 contamination with exposure to a ^{60}Co γ-source, have been off-line processed to obtain the corresponding *scintillation signal shapes* according to the procedure defined in [17].

By a fitting procedure of the signal shapes, the long-lived scintillation lifetime τ_T value has been determined. In Fig.5 [Right] three signal shapes (runs at 0 ppm, 0.6 ppm and 2 ppm of O_2 contamination) are shown with the fitting functions superimposed.

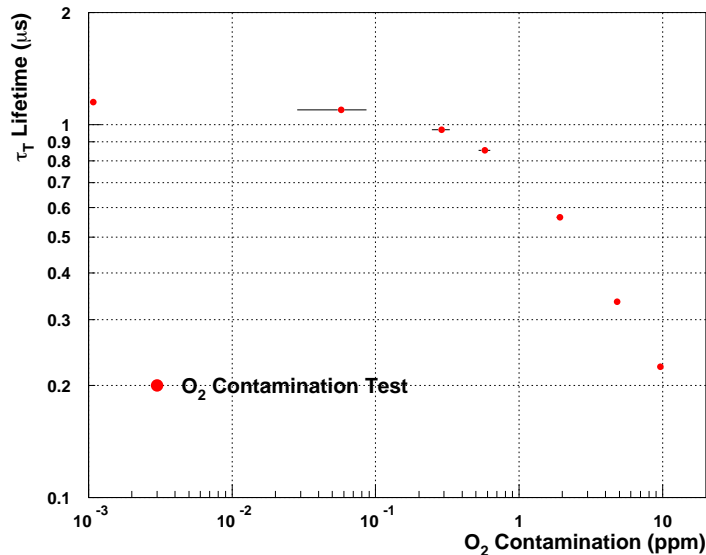The τ_T values from the fit of each run are reported in Fig.3.

Fig. 3. *Long-lived scintillation lifetime at the various [O_2] contaminations (from 0.7 lt detector data).*

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The data in Fig.3 show a clear decreasing behavior of τ_T as the O_2 contamination exceeds the ~ 100 ppb level, due to the quenching process of Eq.4.

The lifetime of the fast component (τ_S), also determined by the fitting procedure of the signal shape, is instead almost unaffected even at the largest contaminations.

4.2   Signal Amplitude Analysis

The signal amplitude is obtained by single waveform integration. This amplitude, expressed in photo-electron units, is proportional to the energy deposited by electrons from ^{60}Co source γ-conversion. Pulse amplitude spectra (mainly from Compton scattering) have been obtained for each run at different $[O_2]$ value. These spectra result to be progressively down-scaled at increasing O_2 concentrations as due to the quenching process (4).

A dedicated fitting procedure has been developed to determine the quenching *scale factor*[5] Q_F relative to each O_2 contamination: the individual entry values of the uncontaminated (0 ppm) spectrum are down-scaled by Q_F, free parameter, to best fit the experimental $[O_2]$ contaminated spectra. The Q_F values thus obtained are reported in Fig.4 as a function of the O_2 contamination.
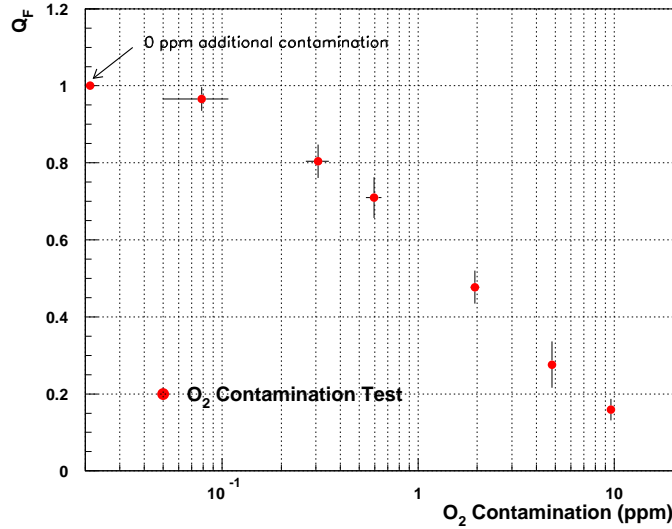

Fig. 4. *Quenching factor Q_F behavior at incremental O_2 contaminations from pulse amplitude spectra analysis (^{60}Co runs with the 0.7 lt test detector).*

A sensitive reduction of the scintillation light yield is found in this high

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[5] The quenching factor Q_F can be equivalently defined as the ratio between the total intensity of scintillation light emitted for a given O_2 contamination with respect to the case of pure liquid Argon.

concentration range ([O$_2$]$\geq$ 100 ppb), e.g. a loss of $\sim$ 40% of the light available from scintillation is accounted to the presence of 1 ppm of O$_2$ in Argon. This confirms that the use of appropriate O$_2$ purification systems is needed also for LAr detectors based on the collection of the scintillation light.

5 Combined analysis of the long-lived component lifetime

In the present study the long-lived lifetime τ_T characterizing the scintillation signal shape in LAr has been measured over a wide range of O$_2$ concentrations, spanning from $\sim$10^{-3} ppm (WArP 2.3 lt data, e.g. Fig.5 [Left]) up to $\sim$10 ppm ("contamination test" with the dedicated experimental set-up, 0.7 lt LAr detector, e.g. Fig.5 [Right]). All the τ_T measurements are reported together in
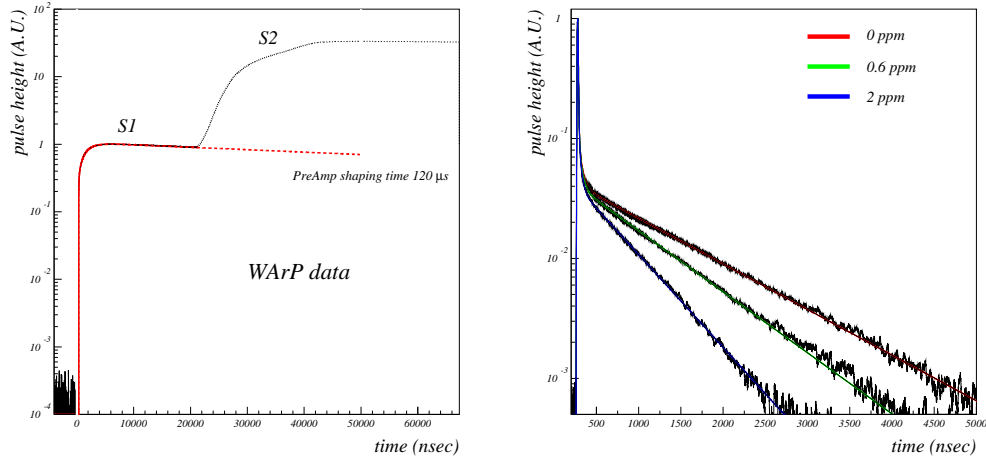

Fig. 5. *Examples of (averaged) signal waveforms collected with the WArP 2.3 lt prototype [Left] and with the 0.7 lt test detector [Right].*

Fig.6. An overall χ^2-minimization, using Eq.6 as fitting function (green curve in Fig.6), allows to extract the following results:

- the rate constant of the quenching process (Eq.4):
  $k'(O_2) = 0.54 \pm 0.03 \; \mu s^{-1} \; ppm^{-1}$;
- the slow-component lifetime at 0 ppb of Oxygen equivalent concentration:
  $\tau_T = 1.21 \pm 0.01 \; \mu s$;
- the initial O$_2$ concentration in the 0.7 lt detector run:
  $[O_2]_{in} = 65 \pm 15$ ppb
  (this value is obtained by using an additive free parameter in the fitting function: $[O_2] \rightarrow [O_2] + [O_2]_{in}$).

The errors associated to the results are from the overall fit (statistical), taking into account the statistical errors from the fit of the signal shapes at different O$_2$ contaminations and also the error associated to the injected amounts of Oxygen in the contamination procedure.

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The fitting behavior (green curve in Fig.6) compared to data shows an increasing discrepancy at higher O_2 concentrations, as possibly due to incomplete absorption of impurities into the liquid. This trend might be explained by a *saturation* effect of the solute (Oxygen) in the LAr solvent. Similar indications have been reported by other groups [14]. The relatively short time scale (hours to day) of the measurements performed prevents to draw any definite conclusion on long term effects.

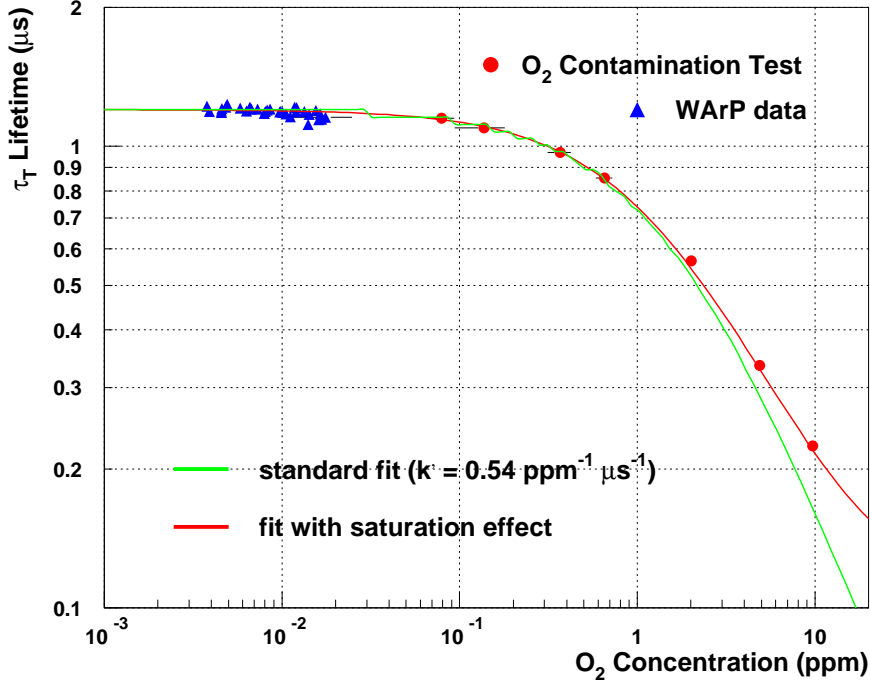A different parameterization of the fitting function for the slow component

Fig. 6. *Long lived scintillation lifetime τ_T vs O_2 concentration from the combined analysis of the WArP 2.3 lt prototype and of the 0.7 lt detector data.*

lifetime has been used, depending on the O_2 concentration under saturation hypothesis:

$$\frac{1}{\tau_T'} \;=\; \frac{1}{\tau_T} \;+\; k'\,\beta\,(1\,-\,e^{-\frac{[O_2]}{\beta}}) \tag{7}$$

where the β parameter represents the concentration scale where saturation becomes effective ($\beta = 13$ ppm from fit) and at first order it reduces to the original fitting function (Eq.6).

The red curve in Fig.6 shows the result of the fit with the modified parameterization. This model considerably improves the scaling with O_2 concentration and decreases the overall χ^2, without changing at all the results reported above.

The goodness of the overall fit (saturation model) is satisfactory (C.L.$\simeq$90%).

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Residual concentration of non-electronegative contaminants in LAr cannot be excluded, i.e. [N$_2$] up to a maximum level of 0.5 ppm (according to the specifications of the 6.0 GAr used for filling in both experimental set-up's here employed). Scintillation light quenching process in LAr due to Nitrogen contamination is in fact known to be rather effective (e.g. with rate constant $k(N_2)$=0.11 μs^{-1}ppm^{-1} [16]). This allows to compute an upper limit for the long-lived component lifetime assuming the N$_2$ concentration at its maximum level. The range of variability is thus defined as 1.21 μs $\leq$ τ_T $\leq$ 1.28 μs.

A test dedicated to the Nitrogen effect on LAr scintillation light has been successively performed with the 0.7 lt detector. The rate constant $k(N_2)$ and the actual Nitrogen content in the 6.0 Ar have been measured as reported in [17].

6 Conclusion

Free electron charge separation and light emission are the two features that characterize the use of LAr as active medium for ionization events.

Residual content of Oxygen in LAr may impair the detector performance by significantly reducing the amount of both charge and light available.

Detailed studies of the consequences of Oxygen contamination on ionization electrons have been reported in literature, showing dramatic effect even in extremely low concentrations as a result of its high electron affinity. This led to the development of appropriate purification systems, key element in the LAr-TPC technology.

The effects on scintillation light have been however less investigated. Residual O$_2$ contamination leads to a substantial reduction also of the scintillation light intensity. This is due to a quenching (i.e. non-radiative) process in two-body collision of O$_2$ impurities with Ar$_2^*$ excimer states, in competition with the de-excitation process leading to VUV light emission.

Two detectors have been used for a detailed test, here reported, of the effects of O$_2$ concentration in LAr: the WArP 2.3 lt prototype and a small (0.7 lt) dedicated detector, coupled with a system for the injection of controlled amounts of gaseous Oxygen.

With the 0.7 lt detector the reduction at increasing O$_2$ concentration of the long-lived component lifetime of the scintillation light emission has been studied, while data from the WArP prototype were used for determining the behavior of both the ionization electron lifetime and the scintillation slow-component lifetime during the O$_2$-purification process activated in closed loop during the acquisition run. The electron lifetime measurements allow to infer the O$_2$ content of the Argon and correlate it with the long-lived scintillation lifetime data.

The effect of Oxygen contamination on the scintillation light has been thus measured over a wide range of O$_2$ concentration, spanning from $\sim$10^{-3} ppm up to $\sim$10 ppm. The rate constant of the light quenching process induced by

Oxygen in LAr has been found to be $k'(O_2) = 0.54 \pm 0.03$ $\mu s^{-1} ppm^{-1}$. This value is large, e.g. compared with the corresponding value of the Nitrogen rate constant, indicating that the use of appropriate O_2 purification systems is needed also for detectors based on the collection of the LAr scintillation light.

7   Acknowledgments

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